GaN Semi-Annual Technical Report for Additional Funding

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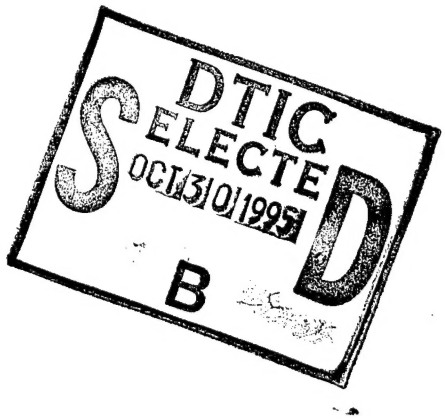

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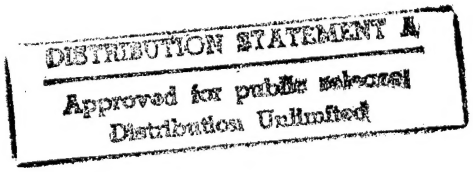

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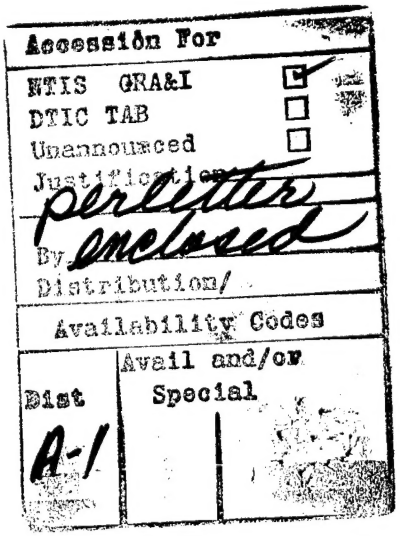

I. Introduction

The superior physical properties[1-2] of gallium nitride (GaN) translate into a potentially very useful semiconductor material for various electronic and optical devices requiring a wide bandgap energy, high breakdown fields, high melting points, high thermal conductivity and high saturation velocities, etc. The recent breakthroughs in GaN-based materials (InGaN/GaN double heterojunction structure), such as commercially available blue LEDs, greatly enhanced the driving force of III-V nitrides, particularly GaN research due to its direct bandgap and promising optical properties.

Theoretically, by changing the composition of group III atoms the quaternary compound InGaAlN will result in material with a direct bandgap ranging from 2.0 to 6.2 eV, covering wavelengths from 600 nm (red) to 200 nm (ultraviolet). This spectral range covers many consumer and military optical devices, including lasers and photo detectors. As an example, the rapid development of GaN for commercial applications such as blue LEDs sends a strong message that the blue laser application by nitride-based materials will be coming soon.

High quality III-V nitrides films have been achieved by several advanced epitaxial techniques. So far, MOCVD technique has produced the best quality GaN thin films due to significant advances in the MOCVD process. The use of a low temperature grown AlN buffer layer[3-7] improved the crystallinity, electrical and optical properties. A room temperature background electron concentration of 1×10^{17} cm^{-3} and mobility of 350 - 400 $cm^2/V\text{-}s$ for a GaN film was obtained. Alternatively, using a GaN buffer layer grown at a low growth temperature at atmosphere pressure further improved the background concentration to 4×10^{16} cm^{-3}, mobility to 600 cm 2/ V-s, and also achieved p-type doping[8-13] of 3×10^{18} cm^{-3}. In Japan, the DH structure blue LEDs became commercially available.

Starting January, 1994, EMCORE has concentrated on the development of multiwafer scale MOCVD processing of GaN in a Turbo-disc reactor. The great effort has been made on the system modification and growth parameters study. As a result, the Turbo-disc reactor becomes a very reliable tool producing large area deposition of GaN films. With the research progress in terms of growth chamber design, heater design and coating, wafer carrier design and coating, we have achieved reproducible GaN thickness uniformity of ~ 2%, background carrier concentration of low $10^{17}/cm^3$ and electron mobility of 300 $cm^2/V\text{-}s$, FWHM of GaN (0002) of 350 arc-sec.

In addition, room temperature photoluminescence (PL) shows a strong band edge transition at 364 nm and two weak shallow state peaks at 550 nm.

II Recent GaN research results

After the first nine month, EMCORE has greatly improved the life time of the filaments, the reproducibility of the GaN films. As a result the high quality p-type GaN thin films have been epitaxially grown on c-sapphire substrates by MOCVD technique in the commercially available multi-wafer rotating disc reactor at 1040°C with a GaN buffer layer of ~200Å at 530°C. The improved undoped GaN films show a n-type background carrier concentration of ~5 x 10^{16} cm^{-3} with an $FWHM_{GaN(0002)}$ of 280 arc-sec across the 1" substrate. The p-doped wafers remain an excellent surface morphology by using CP_2Mg as the p-doping precursor. In addition, after post annealing in N_2 ambient at ~700°C for 20 min to an hour, the Hall measurements show 6.7 x 10^{17} - 1 x 10^{18} cm^{-3} carrier concentration with a hole mobility of 10 cm^2/ v-s which is comparable with the best mobility reported to date. The SIMS depth profile shows an uniform Mg concentration of 6 x 10^{19} cm^{-3} across a 2 μm GaN films, which indicates more than 1% of Mg was activated after the post annealing.

The GaN deposition experiments were carried out in a fully computer-controlled, multi-wafer, rotating disc reactor, MOCVD system equipped with a double wall water-cooling stainless steel chamber, a loadlock, etc. The system has been successfully used [14] to grow crystalline undoped GaN on c-sapphire utilizing trimethylgallium (TMG) and ammonia (NH_3, 100%) as the primary chemical precursors. The gas dynamics of the high speed, low pressure, rotating disk provides the uniform epitaxial growth over a large area. During the deposition process, the wafer carrier rotates at high speed (500-1000 rpm), and the mixed reactants with a main hydrogen flow is introduced from the top of the reactor, which combine to generate a very sharp temperature field over the entire wafer carrier. This approach provides a unique advantage to control GaN two dimensional growth, which in turn results in large area, high quality GaN uniform growth.

In preparation for GaN p-doped epitaxial growth, the c-sapphire substrates first undergo *ex situ* cleaning by HCl, HNO_3 and H_2O. This is followed by *in situ* cleaning in a H_2 flow at 1070 °C. Next is the GaN buffer layer growth in order to overcome the large lattice mismatch between the GaN and c-sapphire. A NH_3 flow of 4-16 l/m and TMG flow of 10-24 sccm were used to produce a 200~300Å layer of amorphous or polycrystalline GaN at 530°C. This thin

GaN layer is crystallized by ramping up temperature process. Finally, p-type GaN epitaxial films are grown by the reaction of NH_3 and TMG at 1040 °C with a flow rate of 4-16 l/m and 24-48 sccm, respectively. The pressure was at 30-200 Torr, and the rotating speed was 500-1000 RPM during the growth process. The biscyclopentadienyl magnesium (Cp_2Mg) was used as p-type doping precursors. Before the p-type doping, an undoped GaN film growth was achieved with a background carrier concentration of $\sim 5 \times 10^{16}$ cm^{-3}. The process diagram is displayed in Fig. 1. A growth rate of 2 ~ 3 μm/h was achieved during the growth cycle. The films were transparent and featureless with a uniformity of ~2% across a 1" substrate.

Several analysis and characterization techniques were utilized to study the GaN films. Optical microscopy and scanning electron microscopy (SEM) were used to study the surface morphology, defects, and growth rate. X-ray diffraction (XRD) and double crystal X-ray diffraction (DCXRD) were practiced to investigate the crystal structure and crystallinity. Transmission electron microscopy was applied to study the interface and defects. The photoluminescence (PL) was employed to investigate the bandgap and emission line. The Hall measurements provided the electrical properties of the grown films.

Fig. 2a contains a Nomarski micrograph of Mg-doped GaN sample, which shows the smooth surface morphology under 400x magnification. The film is transparent and featureless across a 1x1" wafer. The smooth surface morphology was obtained at a wide range of Cp_2Mg flow (30 - 700 sccm). The surface becomes cracked along the crystal direction as shown in Fig. 2b with a high Cp_2Mg flow. The cracking is greatly improved by controlling the cool down process. Under SEM the surface is also smooth as shown in Fig. 2c. In addition, the film thickness is quite uniform across the entire 1x1" wafer, Fig. 2d. The growth rate is slightly reduced with an increased flow of Cp_2Mg.

High resolution transmission electron microscopy (HRTEM) is a powerful tool to study material structure and crystal quality. Fig. 3 is an atomic scale photomicrograph of a undoped GaN epi-layer (including the GaN buffer layer) grown on a sapphire substrate. The well-defined crystal structure of both materials is visible. The GaN layer thickness is measured to be ~200Å, and the boundary between the epi-layer and buffer layer is almost indiscernible. As found by other researchers, stacking faults cross the epi-layer in the vertical direction, caused by the large lattice mismatch between film and substrate. The double crystal X-ray diffraction (DCXRD)

spectrum shows a FWHM of GaN (0002) about 330 arc-sec with a Hall mobility ~ 350 cm^2/ V-s and a carrier concentration of 3 x 10^{17} cm^{-3} across the entire 1x1" substrate.

For the photoluminescence experiments a Spectra Physics argon-ion laser (275-305 nm output wavelength, 750 mW maximum CW power) was used as the photo-excitation source. Typically about 50 mW of this broad-band ultraviolet (UV) laser beam was focused on to the sample. the emission from the sample was focused on to the slits of a ISA HR-640 spectrometer equipped with a special grating sensitive to UV-visible wavelengths. The emission was detected using a RCA 83010E photomultiplier that was connected to a Kiethley 617 digital electrometer. The electrometer was interfaced to a Hewlett-Packard 9000 computer for computerized data acquisition and analysis.

The photoluminescence spectrum of an undoped GaN sample is shown in Fig. 4a. The spectrum is dominated by the characteristic band-edge emission at 3630 Å (3.42 eV) at room temperature. The full width at half maximum (FWHM) of the band edge emission is about 40 meV at 300K which is comparable to the best linewidths[15] from high optical quality GaN layers. The double crystal X-ray diffraction (DCXRD) spectrum shows a FWHM of GaN (0002) about 330 arc-sec with a Hall mobility ~ 350 cm^2/ V-s and a carrier concentration of 3 x 10^{17} cm^{-3} across the entire 1x1" substrate. The characteristic emission spectrum of Mg doped p-type GaN at 300K is shown in Fig. 4b. The spectrum is dominated by an intense, violet, emission band peaking around 4275Å (2.9 eV). The emission is clearly visible to the naked eye at room temperature. The FWHM of this violet peak is approximately 350 meV which again is comparable to the narrowest linewidths[9] from highly p-type Mg-doped GaN. We believe that this intense, narrow linewidth, violet emission at room temperature is clearly indicative of the high optical quality of our p-type, Mg-doped GaN films. The resistivity of p-GaN was measured as 1 ohm-cm.

Fig. 5 contains a secondary ion mass spectroscopy (SIMS) profile of Mg-GaN film before the post annealing. The Mg doping level is 6 x 10^{19} atoms/cm^3 uniformly through the film thickness. To obtain the average carrier concentration in the Mg doped GaN films, Hall measurements were performed at 25°C using alloyed (420 °C, 3 min) Hg-In contacts in a Van der Paw geometry. The ohmicity of these contacts was checked by the linearity of their current-voltage characteristics. For a 2.1 μm thick Mg-GaN sample, we obtained a hole density, p, of 6.67 x 10^{17} cm^{-3} with mobility of 10 cm^2 / V-s. The p-type nature of the GaN was confirmed

by a hot probe test. From the temperature dependence of the carrier density, we obtained an activation energy of ~ 155 meV, consistent with past measurements on Mg-doped GaN[16].

In conclusion, the p-type GaN films have been grown on c-sapphire by MOCVD technique in a production scale multi-wafer rotating disc reactor. The p-type GaN materials have a doping concentration range from 1 x 10^{17} to 1 x 10^{18} cm^{-3} with a hole mobility of 6-10 cm^2/V-s which is comparable with the best reported to date. The SIMS profile shows uniform Mg doping concentration (6 x 10^{19} cm^{-3}) in the film corresponding to 6.7 x 10^{17} cm^{-3} doping concentration from Hall effect indicating more than 1% species were activated. In addition, the p-type epitaxial layer has a good surface morphology, uniformity and crystallinity.

III. Reasons for additional funding

The rapid development of III-V nitride thin films has shown great promise as the basis of a new device technology. Many U.S. Government agencies requires UV/visible light sources, solar blind UV and visible detectors, ultra high-power devices and processors for future system applications. The availability of high quality InGaN/GaN materials is a stimulant to the development of device technology using III-V nitride thin films and is, therefore, of great importance to both the federal government and industry.

In our Phase II program, we have emphasized GaN growth and post device applications such as LED by p type ion implantation, and FET. We will accomplish that with our great effort. In addition, The recent exciting results prove we can go one step further. i.e. we are capable of producing n- and p-type GaN material by epitaxy technology. This makes the device applications field wide open and implicates high temperature electronic devices and blue LED's by InGaN/GaN double heterojunction (DH) structure. With the additional Phase II funding, Success in this LED fabricated in production scale MOCVD predominated program will aid in the effort to put the U.S. in a leadership position in the important research area of deposition of III-V nitride thin films and compete with advanced Japanese LED technology. EMCORE is specialized in epitaxial growth systems and thin film epitaxy technology which are both fundamental components for III-V nitride material technology. Over the past several years, EMCORE has established itself as a major global source of advanced epitaxial growth systems. This major focus has been supplemented by an expanding in-house material capability in GaAs/GaAlAs heterostructures. Many of the major organizations in the compound

semiconductor industry are our customers. The additional funding will also attract the cooperation between EMCORE and the major LED production industry. The extensive interest in GaN/InGaN structure is a major factor in EMCORE's decision to develop a novel GaN growth MOCVD reactor for this emerging technology.

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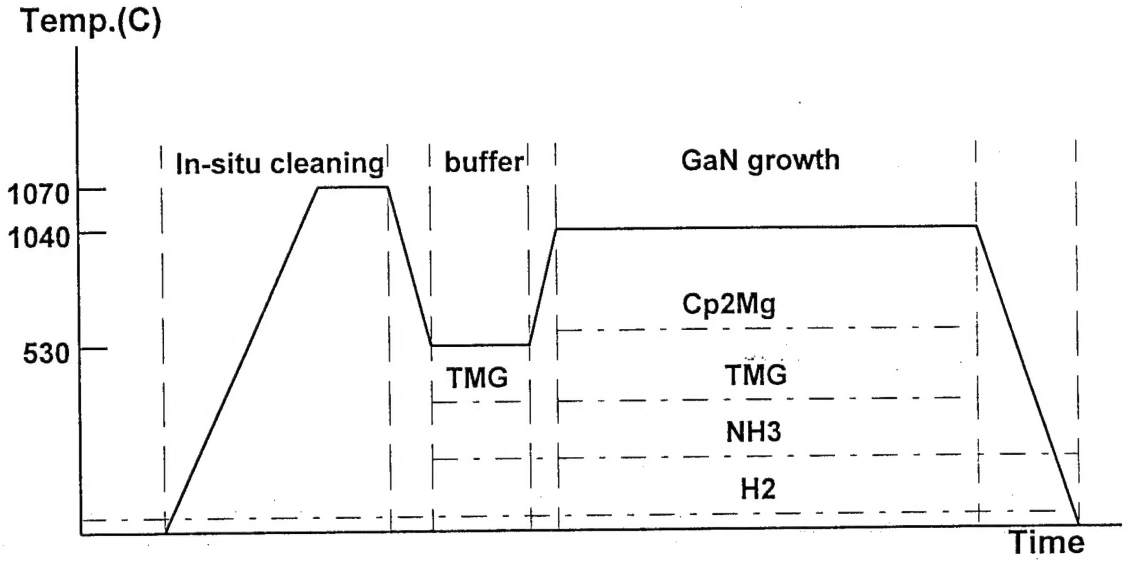

Fig. 1 Process diagram of GaN epitaxy.

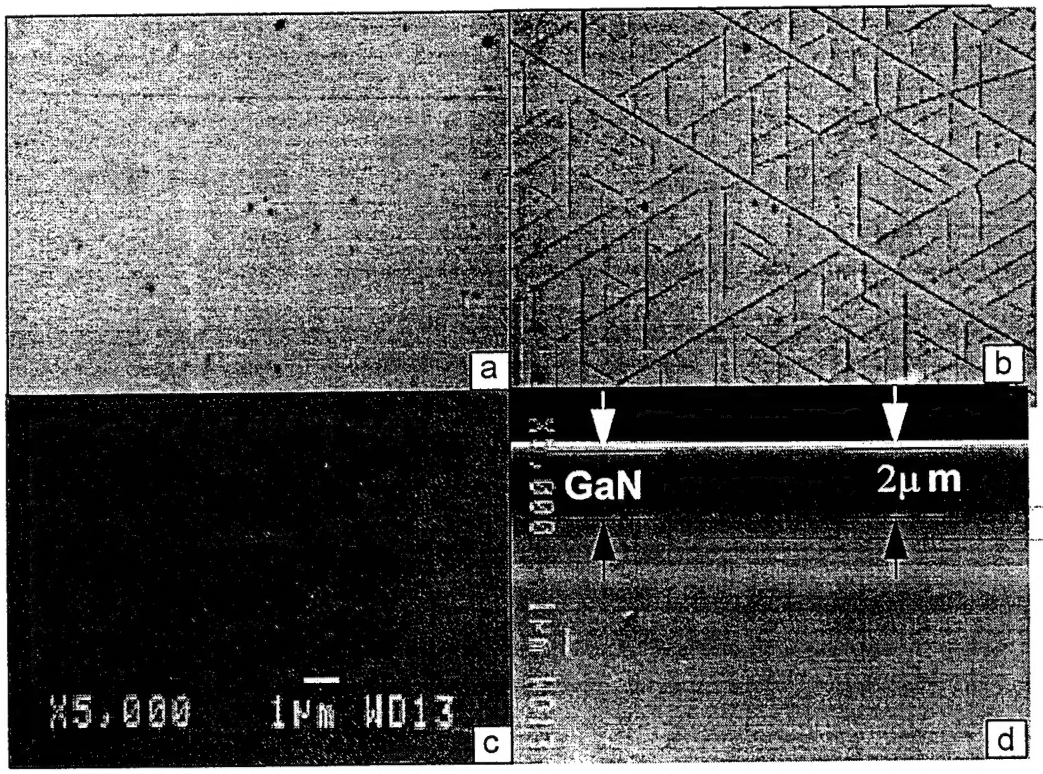

Fig. 2 Nomarski micrograph (400x) of plan-view GaN film.
a) with low Cp_2Mg flow; b) with high Cp_2Mg flow.
SEM micrograph. c) plan-view of GaN; d) Cross-section of p-GaN film on sapphire.

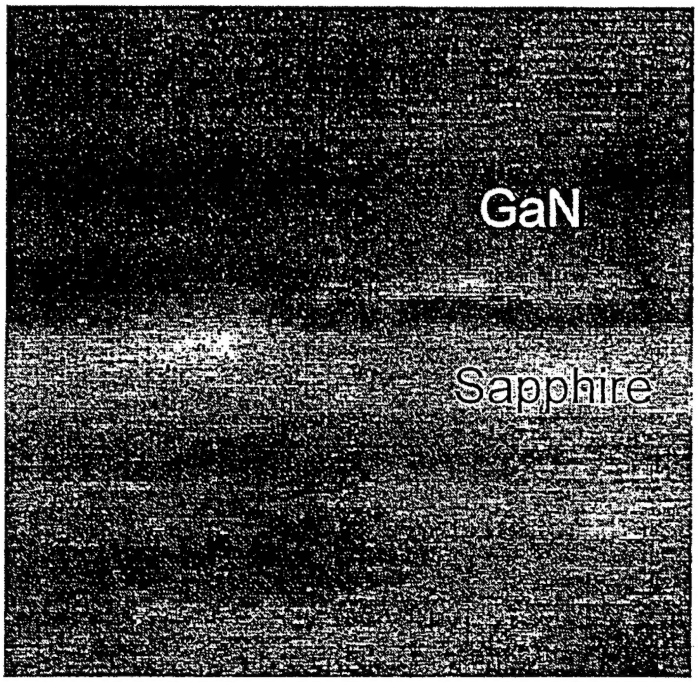

Fig. 3 HRTEM of undoped GaN epitaxial films.

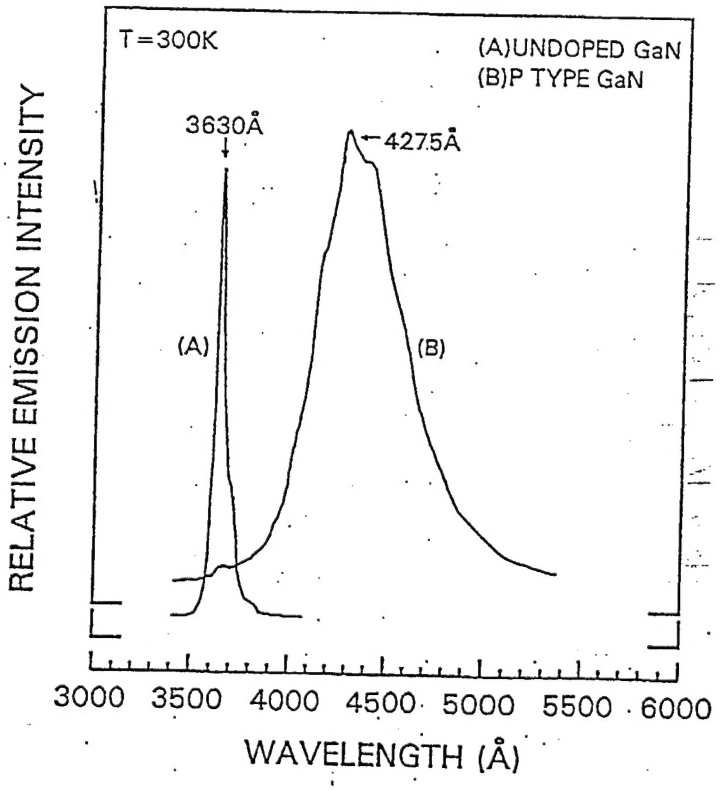

Fig. 4 Room temperature photoluminescence. a) undoped GaN; b) p-GaN.

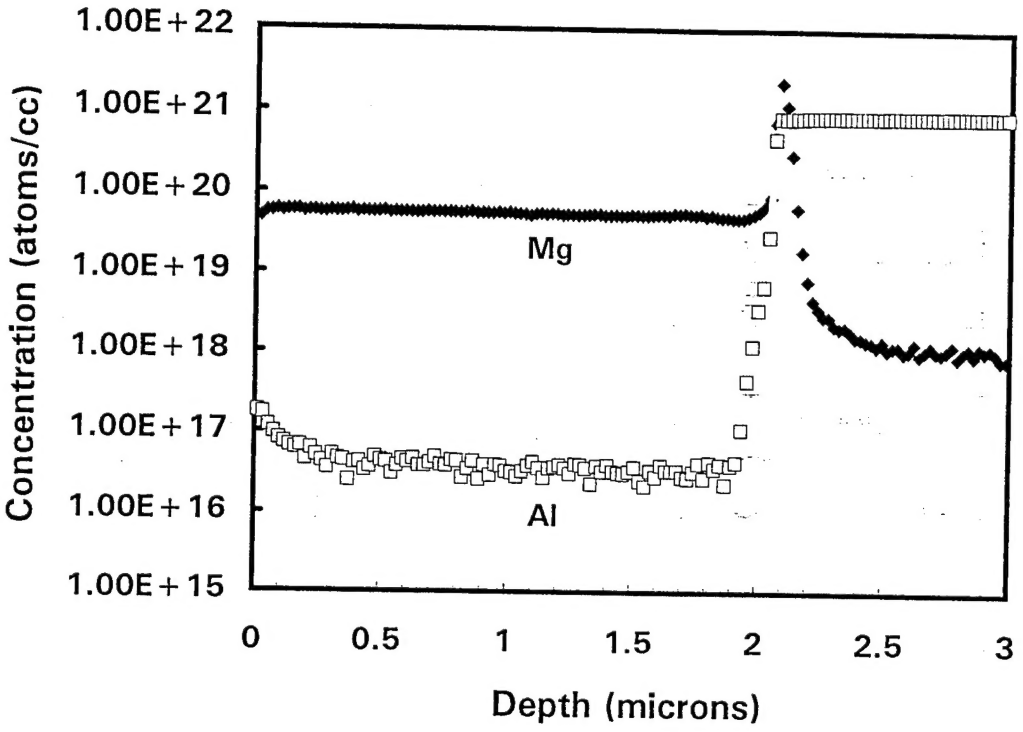

Fig. 5 SIMS profile of Mg-GaN film.

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